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Total Material Variables Influencing a Ductile-Brittle  
Transition Temperature for Molybdenum

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TOTAL MATERIAL VARIABLES INFLUENCING A  
DUCTILE - BRITTLE TRANSITION TEMPERATURE FOR MOLYBDENUM

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Summary

The ductile-brittle transition temperature for a coarse grained recrystallized molybdenum material was found to be lower than for the original fine grained metal from which it was produced. The coarse grain size resulted from a critical straining and subsequent annealing treatment at a low recrystallization temperature. It is concluded from an analysis of various strength parameters which are either measured or estimated for this material that an improved grain boundary strength and a considerable influence of the dislocation substructure are responsible for the less brittle behavior of the coarse grained molybdenum.

Zusammenfassung

Der Spröd - Duktil - Übergang eines grobkörnigen Molybdäns liegt, entgegen herkömmlichen Ansichten, bei einer tieferen Temperatur als der des feinkörnigen Ausgangsmaterials, wenn das Grobkorn durch kritische Verformung und nachfolgende Rekristallisation bei einer niederen Temperatur gezüchtet worden ist. Eine Analyse der Festigkeitskenngrößen zeigt, daß eine erhöhte Korngrenzenfestigkeit und Kleinwinkelkorngrenzen verantwortlich für die erhöhte Duktilität des grobkörnigen Molybdäns sind.

TOTAL MATERIAL VARIABLES INFLUENCING A DUCTILE-BRITTLE  
TRANSITION TEMPERATURE FOR MOLYBDENUM

Introduction

The grain size dependence of a ductile-brittle transition temperature (DBTT) which is observed for steel and similar metals has been described in some detail by Armstrong.<sup>(1)</sup> For recrystallized polycrystals having varying average grain diameters,  $\ell$ , a plot of the DBTT, determined for instance by tensile or by Charpy-v-notch tests, versus  $\ell^{-1/2}$  should be represented by a smooth curve of gradually changing negative slope. The dependence is such that a coarsening of the grain size should result in an increased DBTT. However, it was pointed out in a previous study<sup>(2)</sup> on sintered molybdenum (of chemical analysis given in Table I) that an increase in the polycrystal grain size did not necessarily result in an increased DBTT. Depending on the way of producing the coarse grain size, a deterioration of the transition behavior as well as an improvement could be expected. Figure 1 shows that coarse grained molybdenum, produced by critical straining to approximately 25% and by subsequent recrystallization at low temperatures, exhibits a lower DBTT than the original fine grained metal. Similar results have been reported by other investigators<sup>(3)</sup> who have used the same method for increasing the grain size, but a completely satisfactory explanation for these results has not yet been given.

There have been several attempts to describe the complete ductile-brittle behavior and its dependence on grain size. According to Cottrell<sup>(4)</sup>, the condition for the transition is given by

$$\begin{aligned}
 & (\sigma_0 \ell^{1/2} + k_y) k_y = C \mu \gamma \\
 \text{or} \quad & \sigma_y k_y \ell^{1/2} = C \mu \gamma \quad (1)
 \end{aligned}$$

where  $\sigma_0$  and  $k_y$  are defined as experimental constants from the dependence of the yield stress,  $\sigma_y$ , on  $\ell^{-1/2}$  at constant temperature and strain rate in the Hall-Petch relation:

$$\sigma_y = \sigma_0 + k_y \ell^{-1/2}, \quad (2)$$

$\gamma$  is the effective surface energy of a crack,  $\mu$  is the shear modulus, and  $C$  is a numerical constant. Due to the temperature dependence of the yield stress,  $\sigma_y$ , with all the other terms assumed to be temperature independent, it is possible to calculate a "transition temperature" for a given grain size  $\ell$ . For example, the temperature dependence of  $\sigma_y$  may be written in one form as

$$\sigma_0 = B \exp(-\beta T). \quad (3)$$

If,  $\sigma_0$  is regarded to be composed of a thermal ( $\sigma_0^*$ ) and an athermal stress component ( $\sigma_{0\mu}$ ),

$$\sigma_0 = \sigma_0^* + \sigma_{0\mu}, \quad (3a)$$

then

$$\sigma_0^* = B' \exp(-\beta' T). \quad (4)$$

A relation similar to the Hall-Petch equation applies also for the fracture stress  $\sigma_c$ , i.e.

$$\sigma_c = \sigma_{co} k_c \ell^{-1/2}. \quad (5)$$

Fracture occurs usually be cleavage (therefore the index "c"). As the mode of brittle fracture is normally intercrystalline in recrystallized molybdenum, we should rather write

$$\sigma_b = \sigma_{bo} + k_b \ell^{-1/2} \quad (5a)$$

for this fracture strength. It is normally considered that

$$k_c > k_b > k_y > 0. \quad (6)$$

This means that in coarse grains, at the same applied stress, higher local stress concentrations can be expected, which are therefore able to nucleate cracks, or, in other words, the fracture stress in coarse grained metals is lower.

There is a disadvantage involved in using equation (1): it contains  $\gamma$ , which is not well-defined. This was avoided in two formulae given by Armstrong (5) which contain only the above derived and measurable quantities for the condition that  $T_c$  obtains at  $\sigma_y = \sigma_c$ ; thus

$$T_c = \frac{1}{2} \left[ \ln B - \ln \left\{ (k_c - k_y) + \sigma_{co} \epsilon^{1/2} \right\} - \ln \epsilon^{-1/2} \right] \quad (7)$$

$$T_c = \frac{1}{2} \left[ \ln B' - \ln \left\{ (k_c - k_y) + (\sigma_{co} - \sigma_{ou}) \epsilon^{1/2} \right\} - \ln \epsilon^{-1/2} \right] \quad (8)$$

### Results and Discussion

Figure 2 shows the temperature dependence of the main parameter, the yield strength  $\sigma_y$ , for molybdenum having two of the three different grain sizes shown in Figure 1. If we use the experimentally determined DBTT from Figure 1 (the DBTT obtained from bend tests and tensile tests are comparable (2)) and estimate the brittle fracture stresses for intercrystalline fracture by taking them equal to the yield stresses which apply for these temperatures, following Armstrong (5), then it may be seen for the coarse and fine grain size material that

$$\sigma_b(\text{coarse}) > \sigma_b(\text{fine}) \quad (9)$$

which may mean that

$$\sigma_{b_o}(\text{coarse}) > \sigma_{b_o}(\text{fine}) \quad (10)$$

in analogy to conditions given by the Hall-Petch relation if it is assumed that  $k$  is constant and  $> 0$ . However, the inequality (9) may also be directly taken to show that

$$k_b < 0, \quad (11)$$

even though this is not directly meaningful in terms of the theory underlying the Hall-Petch relation. (1)

As equation (11) is not realistic in terms of other grain size effects and the theory for them, something more has to be considered. The Petch

theory the size effect emphasizes the variation of stress concentrations acting in front of slip planes impinging on grain boundaries, assuming that there is no difference in grain boundary structures of various grain size materials. On this basis, it could be argued that the conditions (9) to (11) simply imply that an increased grain boundary strength exists (larger  $k_b$  value) for the particular large grained material, caused by the previous treatment, which together with the experimentally obtained lower values of the yield strength can explain the lower DBTT.

However, some other findings suggest an additional effect. One could take into account the role of newly originated subboundaries in the coarse grained molybdenum because these boundaries could also act as obstacles to slip bands. Actually, a tendency to transcrystalline fracture has been reported by several investigators (2,6) not only in cold-worked but also in recrystallized material and this appears consistent with the concept of obstacles being generated within the coarse grain volumes, or the elimination, perhaps, of an exceptional grain boundary susceptibility to intercrystalline fracture. On the other hand, there seems to be no effective obstacles within the fine grained molybdenum materials as indicated by a predominantly intercrystalline fracture. Also, subboundaries have not been observed in fine grained material, at least, not so far as etch pitting techniques have been applied. (7)

It should be pointed out that, although the data in Figures 1 and 2 indicate that  $k_b > 0$ ,  $k_y$  remains positive, i.e.  $\sigma_y$  (coarse)  $< \sigma_y$  (fine). Plastic deformation is at least not strikingly affected by any subboundaries which may be present.

Analyzing the stress-strain curves by the extrapolation method<sup>(8)</sup>, it is also possible to determine  $k_y$  and its variation with straining. Figure 3 shows according to this method that  $k_y$  is proportional to the amount of strain which occurred before the recrystallization process. The nature of  $k_y$  was explained by Cottrell<sup>(4)</sup> in terms of the creation of new dislocations in front of pile-ups.

There are other models to explain the detailed character of  $k_y$ . According to Wasilewski (9)  $k_y$  is directly dependent on the substructure (increasing with decreasing distance between dislocations) and this could explain the results shown in Figure 3 because a finer dislocation substructure is expected for increasing strains.

This consideration leads to the same explanation as before: a different substructure exists in the metal of larger grain size, which is obtained by straining and annealing. From Figure 3 it is obvious that the straining has had a first order effect on the increase of  $k_y$ . If we made the assumption which is supported by metallographic evidence that the subgrain size is not smaller than the original grain size, then it turns out that  $k_{ys}$  value for the substructure in the coarse grained material is approximately the same as the  $k_y$  obtained for the fine grain sized molybdenum by the extrapolation method.

With this information, it is now possible to estimate the DBTT for these materials by using equations (2) and (3) in equation (7). One important factor which may immediately be visualized to lead to a lower DBTT seems to be the empirical term  $\beta$  in equation (3) because it has a direct influence on  $T_c$  in equation (7). Indeed we obtained within the experimental scatter of the data a value of  $\beta = 3.18 \times 10^{-3} \text{ }^{\circ}\text{K}^{-1}$  for the fine grained molybdenum and  $5.5 \times 10^{-3} \text{ }^{\circ}\text{K}^{-1}$  for the coarse grained material. This would indicate a lower DBTT for the coarse material but a larger difference in the DBTT values than is experimentally observed, if changes of the other parameters are assumed to be of minor influence. However,  $\beta$  is a very complicated term; it contains an athermal contribution and it is not easy to give  $\beta$  a direct physical interpretation. (10)

A similar term,  $\beta'$ , is obtained by analyzing the stress intercept,  $\sigma_{oy}$  in terms of athermal and thermal components.  $\sigma_o$  of the Hall-Petch relationship

is temperature dependent through the thermally activated  $\sigma_0^*$  (equation 3a) and  $\beta'$  is derived from the exponential equation (4) for  $\sigma_0^*$ . In our material  $\beta'$  has been obtained as  $7.9 \times 10^{-3} \text{ }^{\circ}\text{K}^{-1}$  for the original fine grained molybdenum and as  $10.7 \times 10^{-3}$  for the prestrained and annealed coarse molybdenum. This variation in  $\beta'$  is in more reasonable agreement with the change in DBTT which was measured. These values agree with previous values of  $\beta$  reported by Armstrong (5). This should be expected because  $\beta \rightarrow \beta'$  as  $T \rightarrow 0$  and the values of  $\beta$  reported by Armstrong were determined for temperatures near to  $\sim 200^{\circ}\text{C}$ . However it is reported (11) for steel that  $k_y$  is athermal, and only the athermal component,  $\sigma_{0u}$ , of the yield stress is influenced by different impurities. On the otherhand,  $\beta$  and  $\beta'$  are taken as measures of the temperature dependence of the thermal component,  $\sigma_0^*$ , which on the basis of thermal activation analysis, too, should not change due to the production of different grain sizes. Therefore, it is not clear, why there should be any difference in the  $\beta'$  values.  $\beta'$  and  $\beta$  (as well as  $\beta$  and  $\beta'$ ) are probably prone to changes due to variations in interstitial impurity content. A pronounced stress-induced Snoek effect would decrease the temperature dependence of the yield stress, but this is not likely to be expected in molybdenum with its low solubility for interstitials.

It may also be that the different values for  $\beta'$  are due to experimental uncertainty because of the limited temperature range able to be covered and the limited data points in it. This also applies for the variations that were measured for  $\beta'$ . Substituting an average  $\beta' = 9.3 \times 10^{-3} \text{ }^{\circ}\text{K}^{-1}$  and  $\beta' = 705 \text{ kg/mm}^2$  for both grain sizes in equation (8) and replacing  $k_c$  and  $\sigma_{co}$  by  $k_b$  and  $\sigma_{bo}$ , then, any decrease in the DBTT can be formally discussed as a consequence of any one of the following factors, all of which have been included in the foregoing description:

A.  $\sigma_{bo}$  (coarse)  $>$   $\sigma_{bo}$  (fine) according to equation (10). The effect

of decreasing the DBTT due to increasing  $\sigma_{bo}$  should become more pronounced at large grain sizes due to the factor  $\ell^{-1/2}$  in equation (8).

B.  $k_b$  (coarse)  $>$   $k_b$  (fine) according to equation (6) due to increasing  $k_b$  because  $k_y$  (coarse)  $>$   $k_y$  (fine) as in Figure 3. The decrease in the DBTT in equation (8) should be less pronounced at large grain sizes.

C.  $k_b < 0$ ; equation (11). This condition implies a large  $\sigma_{bo}$  at  $\ell^{-1/2} = 0$  and a decreasing  $\sigma_b$  as  $\ell^{-1/2}$  increases. The DBTT increases at an increasing rate as  $\ell^{-1/2}$  increases.

Table 2 gives the changes in DBTT when certain parameters of equation (8) are varied while the others remain constant.

A calculated DBTT at 96°C for the original fine grained molybdenum agrees well with the result from the bend test. A number of cases are given for the coarse grained material. Considering the three conditions mentioned above, we can first exclude condition (C). In (Ca) of Table 2 the assumption that  $\sigma_{bo}$  remains as low as in the original fine material, is not in accordance with the Hall-Petch relation. The result is an increase of the DBTT, anyway, and this is clearly incorrect. Using a larger  $\sigma_{bo}$ , as estimated from Figure 2 (assuming that  $\sigma_{bo} \approx \sigma_b$ ), results, as shown in (Cb), in a decreased DBTT, but it is still too high. Condition (B) could explain a low DBTT, but it rests solely on the assumptions involved in choosing  $k_b$  and again  $\sigma_{bo}$  is too low.

The only reasonable result seems to come from condition (A), where the large  $\tau_{bo}$  taken together with the assumption of subgrain boundaries responsible for blocking slip bands, gives a calculated low DBTT which is comparable with the experimental results. Of course, a combination of two conditions, as shown in Table 2 for condition (D) = (A + B), could possibly explain the same result, but there are similar restrictions as for (B) alone.

Again we have seen the necessity for the concept of subgrain boundaries

( if we neglect the substructure, we end up basically with condition (Cb) ) as obstacles for dislocation arrays, which has been already discussed, but only when taken together with the possibility of an increased grain boundary strength can the result of Figure 1 be fully verified. The possible importance of substructure in contributing to  $\sigma_o$  has already been discussed for other refractory metals, particularly, niobium. (12)

The results of the present study suggest that another distribution of impurities may also be involved in the enhancement of the ductility of coarse grained molybdenum (produced by prestrain and recrystallizing) because the impurities may be segregating to some degree at the subgrain boundaries.

Figure 4 shows several DBTT curves versus  $t^{-1/2}$  which might apply for a given material under varying conditions. For example, a severe change may occur in the material properties which corresponds to moving from one DBTT curve, say, I to II. Any variation of grain size alone, with no change of the grain boundary strength would have only resulted in a shift along curve I ( $X \rightarrow X'$ ). Curve II stands for a material with an increased grain boundary strength. Thus,  $X \rightarrow Y'$  is the change in  $T_c$  when grain growth also occurs.  $X \rightarrow Y''$  is valid when the effect of increased grain boundary strength is combined with a newly created substructure, the effective grain size of which is the size of the subgrains themselves. The subgrains in molybdenum are apparently never much smaller than the original grains with high angle grain boundaries. High temperature annealing might yield curve III for which the material is distinctly more brittle.  $X \rightarrow Z'$  might result from the combined effect of an increase in grain size and a lower grain boundary strength. This effect can be mitigated somewhat when subboundaries become effective ( $X \rightarrow Z''$ ).

To fully understand the nature of the DBTT, it should be necessary to take into account the role of impurities in determining the mechanism of embrittlement.

ment at various grain sizes, as has already been earlier<sup>(2)</sup> proposed. The Hall-Petch relationship, as usually given for b.c.c. metals with low solubility for interstitials, depends not only on the grain size but on the effect of impurities at the grain boundaries, too. This could be the reason for the argument that  $k_y$  values in b.c.c. metals prone to brittle behavior are larger than those for materials not prone to brittleness, as has been stated.<sup>(1,3,13)</sup> However, the fact that  $k_y$  can be large for a coarse grained material with a low DBTT, reduces the credibility of that general explanation. The fact that an increase in grain size can also cause a lowering of the DBTT makes it necessary to emphasize that, apart from the grain size and even the subgrain size, it is necessary to consider the history of previous treatments to which a material has been subjected. The negative  $\ell^{-1/2}$  dependence of the DBTT is only generally valid, when the same method, which does not alter, or at least does not severely alter the substructure and the distribution of the impurities, has been involved in creating a spectrum of grain sizes.

#### Conclusion:

The preceding discussion shows that the ductile-brittle transition temperature can be changed by altering a number of the parameters involved in specifying the yield and fracture stress of a material susceptible to becoming brittle. Using equations (7) and (8) for computing the transition one has to be aware that a number of material parameters may be altered when a material has been treated in any way. However, it should be possible to evaluate these parameters and the changes they produce by performing specific quantitative measurements.

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Figure Captions

Fig. 1. The ductile-brittle transition for recrystallized molybdenum of different grain sizes as observed in a bend test. (2)

Fig. 2. The temperature dependence of the yield stress for recrystallized molybdenum of different grain size. (2)

Fig. 3. The influence of deformation before recrystallization on  $k_y$ . (2)

Fig. 4. The effect of grain size and grain boundary properties on the ductile-brittle transition. Curve I shows the dependence for the original material. Curve II represents a material with higher, curve III one with lower grain boundary strength.

Table Captions

Table I. Average chemical analysis of sintered molybdenum

Table II. Calculation of the DBTT by means of equation (8)

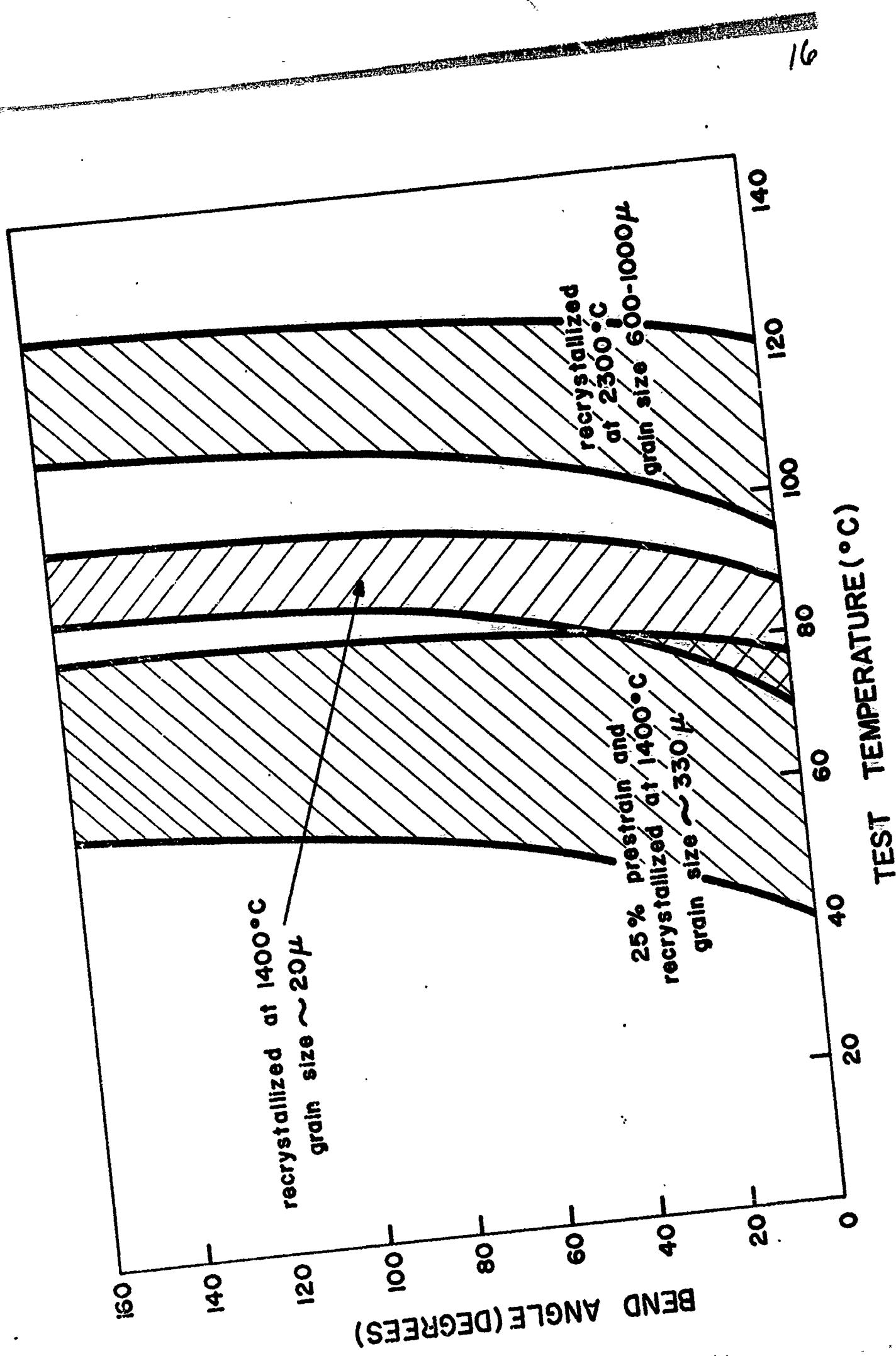
Table 1

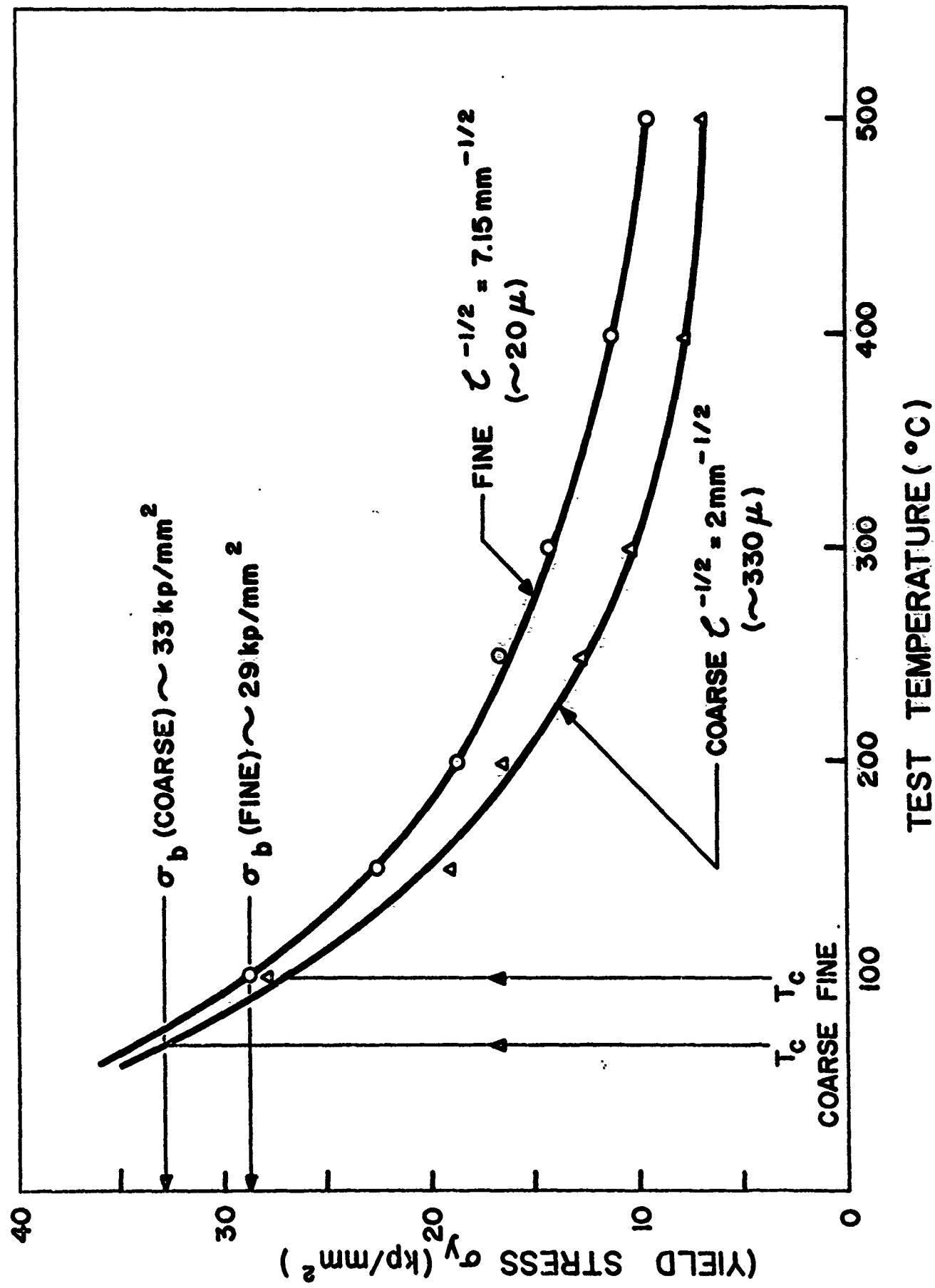
Average Chemical Analysis of Sintered Molybdenum (in wt. ppm)

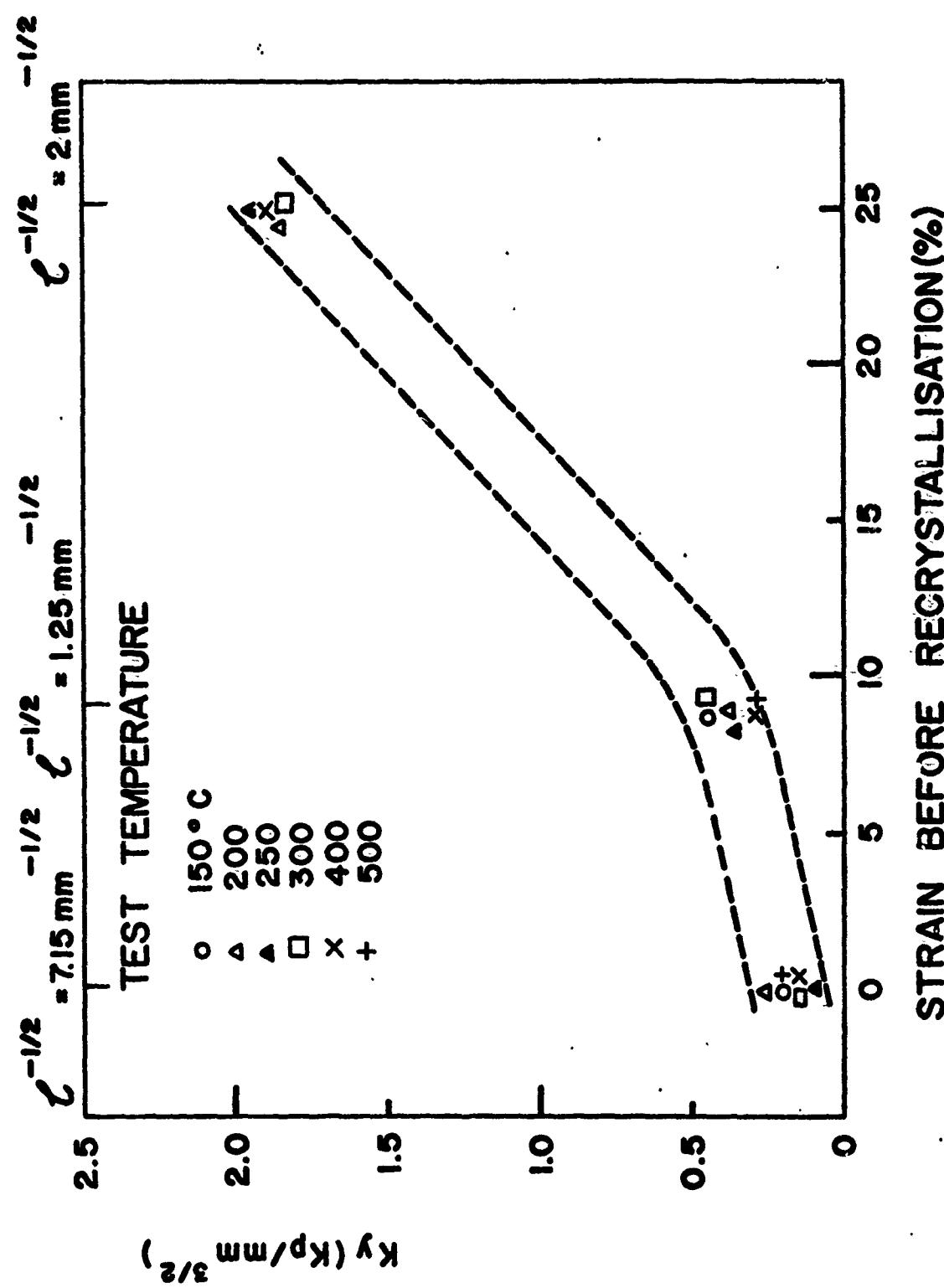
Oxygen	Carbon	Nitrogen	Hydrogen	Tungsten	Iron
50	30	40	10	1000	100

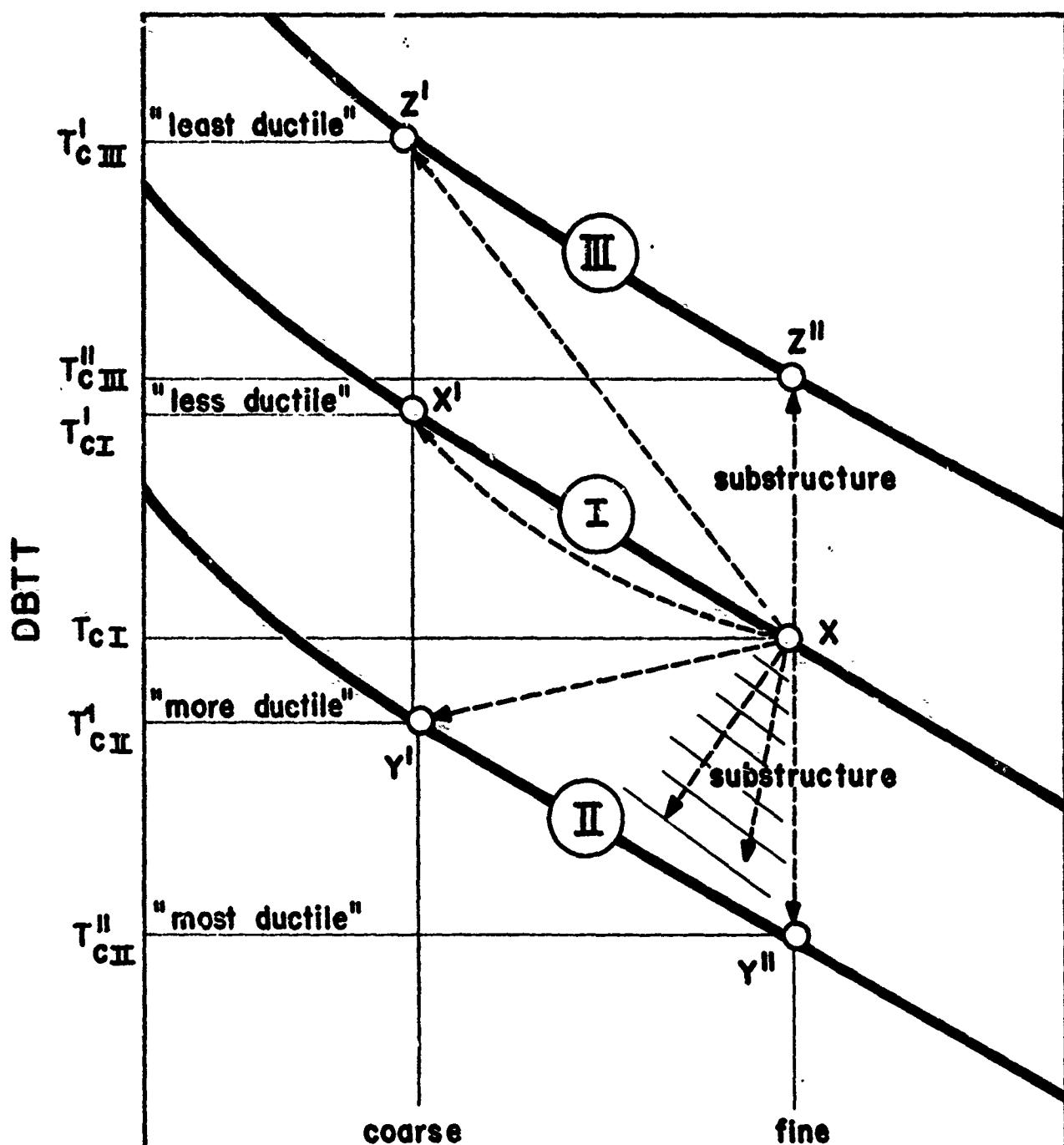
TABLE 2. CALCULATED VALUES OF THE DBTT

GRAIN SIZE	CONDITION	ASSUMPTIONS				DBTT °C	AGREEMENT WITH EXPERIMENT
		$\sigma_{b0}$ kp/mm <sup>2</sup>	$k_y$	$k_b$	SUB- STRUCTURE		
FINE		29	0.33	2 ky	no	$\ell^{-1/2} = 7.15$	96
	(A) $\sigma_{b0}(\text{coarse}) > \sigma_{b0}(\text{fine})$	33	0.33	2 kys	yes	$\ell^{-1/2} = 7.15$	yes
C O	(B) $k_b(\text{coarse}) > k_b(\text{fine})$	29	1.9	2 ky	no	$\ell^{-1/2} = 2.0$	83
	(Ca) $k_b < 0$	29	1.9	-1.0	no	$\ell^{-1/2} = 2.0$	133
A R S E	(Cb) $\sigma_{b0}(\text{coarse}) > \sigma_{b0}(\text{fine})$	33	1.9	-1.0	no	$\ell^{-1/2} = 2.0$	104
	(D) $\sigma_{b0}(\text{coarse}) > \sigma_{b0}(\text{fine})$ $k_b(\text{coarse}) > k_b(\text{fine})$	33	1.9	2.5	yes	$\ell^{-1/2} = 7.15$	73









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